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IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re patent application of:

David J. Robson, et al.

Serial No. 08/876,322

Filed June 16, 1997

IMPROVEMENTS IN OR RELATING TO ABSORPTION OF HYDROPHOBIC WATER-IMMISCIBLE LIQUIDS

Before the Examiner
Ivars C. Cintins

Group Art Unit 1724

I hereby certify that this correspondence is being deposited with the United States Postal Service as first class mail in an envelope addressed to: Commissioner of Trademarks, 2900 Crystal Drive Arlington, VA 22202-3514 on December 22 2003
(Date of Deposit)

James B. Myers, Jr
Name of applicant, assignee, or Registered Representative

James B. Myers Jr
Signature

December 22, 2003
Date of Signature

DECLARATION OF RICHARD SALISBURY
SUBMITTED UNDER 35 USC §131

I, Richard Salisbury, a British subject of Berthlwyd, Tynygongl, Ynys Môn, LL74 8NS, Wales declare as follows.

1. I am making this Declaration in support of US Patent Application No. 08/876,322 (herein referred to as "the Application").

2. I was awarded a Master of Arts Degree in Natural Sciences by the University of Cambridge in 1971. I have been employed in The BioComposites Centre at the University of Wales, Bangor, Gwynedd, LL57 2UW, Wales as a Research Scientist since 1988 specialising in wood modification, particleboard development and large scale chemistry.

3. The BioComposites Centre does have a financial interest in the Application.

4. I have reviewed and am familiar with the Application. I understand that the United States Patent & Trademark Office has rejected the claims of the Application over the teachings of US Patent No. 3,607,741 (Sohnius). I have reviewed Sohnius and am familiar with what Sohnius teaches regarding the preparation of an oil slick removal system.

5. I endeavoured to prepare an oil slick removal material in accordance with the teachings of Sohnius. The structure of such a material is broadly outlined at column 1, lines 43 to 48 of the Sohnius disclosure and is described as:

> "A unitary mass of pieces of finely divided oil absorptive and adsorptive cellulosic material having integral air cells for floatation, resilient fibre content to prevent compaction of the mass, (and) water repellent interspersed throughout to prevent water saturation of the mass".

6. As indicated in the quoted passage, production of the Sohnius material requires a "water repellent". An example of such a repellent is given in the table at column 2, lines 29 to 38 of the Sohnius patent, and I endeavoured to use such a repellent in the present work. I would, however, specifically draw attention to the following points:

(i) The repellent requires the use of "paraffin", but this term has a multiplicity of meanings in different places and contexts, and little further guidance as to the particular form of "paraffin" employed by Sohnius is given in the disclosure of his patent. The term "paraffin" usually refers to a saturated hydrocarbon, and it is clear that it must be non-volatile in order to survive the drying process employed in the procedure for producing the Sohnius material. However, this still leaves a wide range of possible "paraffins" that could be employed. In the present work, I employed "liquid paraffin BP" and a microcrystalline paraffin wax emulsion (available under the designation Mobilcer 538).

(ii) The table in column 2 refers to a "silicone". Examples of such "silicones" are given in the disclosure at column 2, lines 40 to 44 of the Sohnius disclosure. Examples given are dimethyl silicone fluids of which the dimethyl polysiloxanes are examples. For the present work, I chose to use poly(dimethyl silicone) available under the designation "Dimethicone".

(iii) The table on page 2 of Sohnius specifies use of a "stearate". The preferred "stearate" is exemplified in column 2, lines 47 to 46 as being "calcium stearate", and this was employed in the present work.

(iv) The table on page 2 specifies an "emulsifier" for which a preferred example is indicated at column 2, lines 58 to 60 as being 3,5-dimethyl - 1-hexyn-3-ol which was employed in the present work.

7. I encountered a number of problems in endeavouring to produce the Sohnius material. An initial problem was in relation to the production of "water repellent" materials of the type summarised in the Table on page 2 of the Sohnius disclosure as discussed more fully above in paragraph 6above. When all of the ingredients of the water repellent were mixed together in the preferred proportions shown in the Table, they produced a "sticky paste" which was very difficult to mix with wet paper pulp (see the disclosure at column 2, lines 2 to 4 of the Sohnius disclosure). The repellent formed globules rather than coating the pulp fibres. I did, however, find that this problem could be overcome by adding the calcium stearate component of the "water repellent" after the other ingredients.

8. A less tractable problem was due to the fact that paper pulps do not readily form a "pulpy froth" which is one of the required steps in the production of the Sohnius material as disclosed at column 2, line 4. The relatively dry paper pulps form small clumps of pulp with continuous air channels between them. When wetted, the paper pulps can

include air bubbles, but these collapse rapidly when agitation ceases, and the resulting structure is simply one of a three-dimensional pulp network in water.

9. Many formulations were tried within and beyond the ranges suggested in Table 2 of column 2 of the Sohnius disclosure. Different procedures and devices were used to encourage the pulp to take on a cellular structure, but none were successful.

10. The only way that I found to make a "pulpy froth" was to use a foaming agent such as a combination of a surfactant with a foam enhancer such as coconut diethanolamide. This can be added to the pulp before beating or used to make a foam that is incorporated into the pulp mixture. By such methods, a material with the structure described by Sohnius could be produced, but such materials are always so hydrophilic that they sank in minutes when placed on the surface of clean water. In any event, this approach (i.e. use of a foaming agent) is not described in the Sohnius disclosure.

11. I found that if the Sohnius procedure was followed as described, then the resulting material was hard and quite dense as would be expected from dried paper pulp. This was the case when either "liquid paraffin BP" or Mobilcer 538 was used as the paraffin. It was also the case irrespective of whether or not wood fibres were used in conjunction with the paper pulp. The materials <produced is following the Sohnius procedure> were subjected to two tests. The first was to test the ability of the materials to float on clean water since an "oil slick removal system" is advantageously capable of floating on water for some time until it may actually come into contact with oil. In accordance with this test, I found the Sohnius materials to be very hydrophilic and they sank in under 10 minutes. The second test was to evaluate the oil pick-up characteristics of the Sohnius material since Sohnius claims that his material will pick up 6.6 times its own weight in oil (see paragraph bridging columns 2 and 3). In order to test the oil pick-up properties of the materials I had produced, they were dropped onto a continuous slick of oil on water (around 500g/m^2). The oil used for these tests was a thermal oil chosen because its low volatility allowed the materials tested to be oven-dried to remove any water picked up in the test so that the amount of oil picked-up could be determined. I found that some of the materials I had produced were capable of picking-up close to 6.6 times their own weight in oil, but these were not materials that would float in clean water for usefully long periods (see results of the first test).

12. I also prepared modifications of the Sohnius materials by emitting the emulsifier, i.e. 3,5-dimethyl-1-hexyn-3-ol. When tested in clean water, it was found that the resulting materials stayed afloat overnight because the rate of penetration of water was low. After one hour in the water, the parts submerged in the water were wet to a depth of about 6mm. Only the parts that were still dry (i.e. the parts above water) would absorb oil. With this sort of behaviour, it was not possible to carry out a meaningful repeat of the second test detailed in paragraph 11 above.

13. In contrast with the oil pick-up properties of the materials produced according to the Sohnius disclosure, I also tested an acetylated wood fibre that was produced in accordance with the claims and as taught in the present Application using a similar slick to that described in paragraph 11 above. I found that the acetylated wood fibre held in a net picked up 14 times its weight of the oil even after floating on water for 18 hours beforehand. The acetylated water fibre was also much quicker at picking up the oil.

14. I also endeavoured to produce a material comprised of wood fibre (of the type employed in the present Application) and a "water repellent" of the type proposed by Sohnius. This material was prepared by thorough mixing of the following components:

Component	Amount
Wood fibre	10.0 g
Dimethicone	0.10 g
Liquid Paraffin or Mobilcer 538	0.20 g
Calcium Stearate	0.15 g
3,5-dimethyl-1-hexyn-3-ol	0.05 g
Water	60 or 100 g

15. The total amount of additives used is 5% of the weight of fibre, which is at the top of the range recommended by Sohnius above which there is no further improvement.

16. To produce the materials, the Dimethicone, paraffin, calcium stearate, and 3,5-dimethyl-1-hexyn-3-ol were mixed with the water and blended with a high shear mixer. The wood fibre was then stirred in using the mixer at first and then by hand. The wet fibre was then formed into lumps. The samples prepared with Liquid Paraffin (and either 60 or 100 g water) and those proposed with Mobilcer 538 (and 60 g water) were dried at 55°C overnight. The sample prepared with Mobilcer 538 and 100 g water was found to be much too wet with the result that a lot of the liquid ingredients drained out of the sample before drying. Consequently this sample was not pursued further.

17. The dried materials produced in accordance with paragraph 16 were tested by floating lumps thereof on clean water. It was found that all the samples became waterlogged and sank within five minutes.

18. In contrast, material produced according to the claims and teachings of the present Application, as described above, was found to float on clean water for at least 60 hours.

19. I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that wilful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code, and that such wilful false statements may jeopardise the validity of the Application or any patent issuing thereon.

7 November 2003
Date

Signature

Richard Salisbury
Typed Name